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(54) ## CERIUM BASED ABRASIVE MATERIAL, METHOD OF QUALITY EXAMINATION THEREFOR AND METHOD FOR PRODUCTION THEREOF

(57) The present invention provides a method of examining quality of cerium-based abrasives which can simply determine their grinding characteristics. Specifically, the method employs X-ray diffractometry to examine qualities based on, for example, B/A wherein A and B are peak intensities relevant to Ln_xO_y and LnF_3, respectively. The present invention further provides: a method of producing a cerium-based abrasive which can give cerium-based abrasive with specific grinding characteristics; and a cerium-based abrasive which has specific grinding characteristics for specific purposes.

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Description

TECHNICAL FIELD

[0001] This invention relates to a method of examining quality of cerium-based abrasives, a method of producing a cerium-based abrasive, and a cerium-based abrasive.

BACKGROUND ART

[0002] Recently, glass materials have been used for various purposes. Of these, advanced glass materials including glass substrates for optical and magnetic disks, active matrix type liquid crystal displays (LCDs), color filters for liquid crystal TV sets, watches, calculators, LCDs for cameras, displays for solar cells or the like, LSI photomasks and optical lenses, and optical lenses themselves are required to be surface-ground highly precisely.

[0003] These glass substrates are normally surface-ground using a cerium-based abrasive composed of an oxide of rare-earth, in particular cerium oxide, as a main ingredient, because cerium oxide shows several times higher grinding efficiency than zirconium oxide and silicon dioxide for grinding glass materials.

[0004] The common stock materials for cerium-based abrasives include rare-earth materials, e.g., carbonates, hydroxides and oxalates of rare-earth elements, and oxides produced by burning them. These stock materials are normally prepared from bastnasite concentrate or other cerium-containing rare-earth materials by removing a part of rare-earth elements, e.g., neodymium (Nd) and prasceodymium (Pr), and radioactive materials and the like by a known chemical treatment.

[0005] A cerium-based abrasive from a carbonate or oxide of rare-earth is produced by the following process. The process starts with slurrying or wet-crushing the stock material followed by chemical treatment with a mineral acid and, as required, with hydrofluoric acid or ammonium fluoride. The resultant slurry is subjected to filtration, drying and roasting. Finally, it is crushed and classified to have an abrasive of specific particle size.

[0006] A cerium-based abrasive is required to have specific grinding characteristics for specific purposes. It is therefore necessary to grasp the grinding characteristics of a produced abrasive by, e.g., analyzing these characteristics. For example, it is known that grinding speed, referred to as grindability as one of important grinding characteristics, increases as the abrasive crystal size grows by the roasting step. It is however difficult to accurately grasp the grinding characteristics only from average particle size determined by the common method of analyzing particle size distributions. Therefore, an abrasive is examined, as required, for its quality by a test in which a test piece is actually ground by the abrasive. The grinding test is time-consuming, because the ground test piece is weighed to determine grindability, or ground surface is observed to confirm scratches. Therefore, a more simple method of examining the quality has been in demand. At the same time, a method of producing abrasives with specific grinding characteristics can greatly save works for, e.g., quality examination, and hence is desirable, because it produces abrasives more efficiently.

[0007] The present invention is developed in the light of the above problems. It is an object of the present invention to provide a method of examining quality of cerium-based abrasives which can simply determine their grinding characteristics. It is another object of the present invention to provide a method of producing a cerium-based abrasive which can give cerium-based abrasives with specific grinding characteristics. It is still another object of the present invention to provide a cerium-based abrasive which has specific grinding characteristics for specific purposes.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008]

Figure 1 shows the relationship between grindability and C/A ratio. Figure 2 shows the X-ray diffraction analysis data of Abrasive 1. Figure 3 shows the X-ray diffraction analysis data of Abrasive 6.

DISCLOSURE OF THE INVENTION

[0009] The inventors of the present invention have noted incorporation of fluorine (F) in a cerium-based abrasive in order to solve the above problems. Fluorine, which has been incorporated mainly to improve grindability, forms a material of specific crystalline structure, depending on its content and roasting temperature, in abrasive or stock therefore in which it is incorporated. More concretely, a fluorine-containing compound is found to have the following crystalline structure observed by X-ray diffractometry (XRD) in a cerium-based abrasive or stock therefor in which it is incorporated:

(1) A fluorine (F) -containing compound is present in the form of LnF_3 (e.g., LaF_3, wherein Ln is lanthanoids and La is lanthanum) in an F-containing oxide of cerium-based rare earth, and $LnCO_3F$ in an F-containing carbonate

of cerium-based rare earth in stock material before roasting.

(2) When $LnCO_3F$-containing stock material is roasted, Ln having a larger ionic radius decreases in content in a solid solution, being discharged in the form of LnF_3 and contracting the Ln_xO_y lattice. This changes the crystal phase. For example, the crystal phase is transformed from the one identified as $Ce_{0.5}Nd_{0.5}O_{1.75}$ by X-ray diffractometry (XRD) to the one identified as $Ce_{0.75}Nd_{0.25}O_{1.875}$. It should be noted, however, that the crystal phase identified as $Ce_{0.5}Nd_{0.5}O_{1.75}$ or $Ce_{0.75}Nd_{0.25}O_{1.875}$ has the main peak in the XRD pattern, even at a low Nd content, from which it is considered to be an oxide containing La, which is normally present in a cerium-based abrasive at several tens % by atom on Ce, namely on the order of 0.2 to 0.7 times the Ce content in the number of atoms.

(3) LnF_3, when discharged at a high roasting temperature, grows as the LnOF phase (e.g., discharged LaF_3 grows as LaOF phase). Growth of this phase will be retarded as a fluorine content increases, unless a roasting temperature is increased. Conversely, it grows at a lower roasting temperature, as an F content decreases.

[0010] Ln (lanthanoids) in this specification contains at least one element of La (lanthanum), Ce (cerium) or Nd (neodymium) , and LnF_3 is LaF_3, CeF_3 or the like, and LnOF is LaOF, CeOF or the like. Ln_xO_y is La_2O_3, CeO_2, $Ce_{0.5}Nd_{0.5}O_{1.75}$, $Ce_{0.75}Nd_{0.25}O_{1.875}$ or the like, where normally the relationship $3/2 \leq y/x \leq 2$ holds. These compounds generally contain La at 5.0% to 85% per 100% of Ce, namely on the order of 0.05 to 0.85 times the Ce content in the number of atoms, and Nd at 1.0% to 50% per 100% of Ce, namely on the order of 0.01 to 0.5 times the Ce content in the number of atoms.

[0011] The inventors of the present invention have further studied extensively based on the above XRD analysis results and found that whether or not a cerium-based abrasive containing F and, at the same time, La or Nd to some extent has good quality (grinding characteristics) can be determined by its XRD analysis results, reaching the present invention.

[0012] More concretely, the present invention provides a method of examining quality (grinding characteristics) of a F-containing cerium-based abrasive containing La or Nd each at 0.5% or more per 100% of Ce, all by atom, and having a specific surface area of $12 m^2/g$ or less, the method being based on XRD analysis with Cu-$K\alpha_1$ line as an X-ray source, wherein the XRD analysis measures intensity A of the maximum peak "a" in a diffraction angle (2θ) range from 5 to 80°, and at least one of intensity B of the maximum peak having a diffraction angle in a range of 27.5±0.3° and smaller than the angle of the peak "a," intensity C of the maximum peak having a diffraction angle in a range of 26.5±0.5°, and intensity D of the maximum peak having a diffraction angle in a range of 24.2±0.5°, and at least one of the B/A, C/A and D/A ratios is determined, to examine the abrasive by comparing one of the ratios with that of an abrasive of known grinding characteristics determined by XRD analysis.

[0013] When an abrasive from a stock material containing limited quantities of impurities other than rare-earth elements, e.g., carbonate or oxide of rare-earth, is analyzed by XRD, the maximum peak "a" (diffraction angle: $2\theta_A$, intensity: A) in a diffraction angle (2θ) range from 5 to 80° is normally relevant to the [111] plane of Ln_xO_y ($1 \leq y/x \leq 2$). The maximum peak "b" (diffraction angle: $2\theta_B$, intensity: B) having a diffraction angle in a range of 27.5±0.3° and smaller than the angle of the peak "a," if appears, indicates the presence of LnF_3. The maximum peak "c" (diffraction angle: $2\theta_C$, intensity: C) appearing at diffraction angle in a range of 26.5±0.5° indicates the presence of LnOF, and the maximum peak "d" (diffraction angle: $2\theta_D$, intensity: D) appearing at a diffraction angle in a range of 24.2±0.5°, if appears, indicates the presence of LnF_3 as well as peak b.

[0014] It is found that there is a certain relationship between the XRD analysis results (e.g., intensity A of the maximum peak relevant to Ln_xO_y, and B/A and D/A ratios, wherein B and D are intensities of the maximum peaks relevant to LnF_3) and quality (i.e., grinding characteristics related to scratches and grindability) of a cerium-based abrasive. When a relationship between B/A, C/A or D/A ratio and grinding characteristics of a cerium-based abrasive of known grinding characteristics is established, grinding characteristics of another cerium-based abrasive can be simply examined by measuring its XRD characteristics, e.g., B/A ratio. It is found by investigating the results that the above examination method is particularly effective for an F-containing cerium-based abrasive containing La or Nd each at 0.5% or more per 100% of Ce, all by atom, and having a specific surface area of $12 m^2/g$ or less. In particular, the abrasive preferably contains fluorine at 1.0 to 15% by weight. At less than 1.0% by weight, the abrasive may deviate from the range in which it can be adequately examined by the present invention; for example, it may not have the B/A ratio of 0.06 or more even when it produces a lot of scratches. At more than 15% by weight, on the other hand, the abrasives satisfying the examination standard of, e.g., B/A<0.06, will be rarely produced.

[0015] In the actual quality examination, a cerium-based abrasive is analyzed by XDR to measure the peak "a" and at least one of the peaks "b," "c" and "d," and find at least one of B/A, C/A and D/A ratios, and the characteristics of a cerium-based abrasive (grinding characteristics) are examined based on the established relationship between the grinding characteristics and the ratio.

[0016] The quality examination standards include whether the relationships B/A<0.06 or D/A<0.04 or not. It is found that an abrasive leaves only a limited number of scratches on the ground surface, e.g., glass surface, as its B/A or D/A ratio decreases, and that a cerium-based abrasive satisfying the relationship B/A<0.06 or D/A<0.04 produces scratch-

relationships $B/A<0.06$ or $D/A<0.04$ and $0.05 \le C/A \le 0.60$ can be provided as the one having a required practical grindability, because it leaves few scratches on the ground surface. A cerium-based abrasive simultaneously satisfying the relationships $B/A<0.06$ or $D/A<0.04$ and $0.10 \le C/A \le 0.60$ is suitable for primary grinding of glass for liquid crystal and hard disks. A cerium-based abrasive simultaneously satisfying the relationships $B/A \le 0.01$ or $D/A \le 0.008$ and $0.10 \le C/A \le 0.60$ is suitable for finish grinding of glass for liquid crystal. A cerium-based abrasive simultaneously satisfying the relationships $B/A \le 0.01$ or $D/A \le 0.008$ and $0.05 \le C/A \le 0.10$ is suitable for finish grinding of hard disks.

[0025] The cerium-based abrasive of the present invention is normally used in the form of slurry, after being dispersed in a dispersion medium, e.g., water, to 5 to 30% by weight. The dispersion media useful for the present invention include water-soluble organic solvents, e.g., alcohol, polyhydric alcohol, acetone and tetrahydrofuran. However, water is a normal selection.

[0026] The cerium-based abrasive of the present invention preferably contains a high-molecular-weight organic dispersant. The organic dispersants useful for the present invention include polyacrylates, e.g., sodium polyacrylate, and carboxymethyl cellulose, polyethylene oxide and polyvinyl alcohol. Such an organic dispersant works to prevent foaming during the grinding process. It is incorporated in the abrasive at 0.1 to 0.8% by weight, beyond which it will exhibit little further effect.

[0027] The inventors of the present invention have also studied a method of producing an abrasive having required grinding characteristics for a specific purpose, based on the above-described XRD analysis results, to find that at least one of the B/A, C/A and D/A ratios, determined by XRD peak intensities, changes regularly with a fluorine content of the abrasive or temperature at which it is roasted, reaching the invention described below.

[0028] This inventions relates to a method of producing a fluorine-containing cerium-based abrasive containing La or Nd each at 0.5% or more per 100% of Ce, all by atom, and having a specific surface area of 12 m^2/g or less, involving fluorination treatment of the abrasive before it is roasted, wherein a fluorine content and roasting temperature are determined by the XRD analysis results. More concretely, the cerium-based abrasive is analyzed by XRD with Cu-$K\alpha_1$ line as an X-ray source, to determine the maximum peak intensities A, B, C and D in a diffraction angle (2θ) range from 5 to 80°, 27.5±0.3° smaller than diffraction angle of peak a, 26.5±0.5° and 24.2±0.5°, respectively, and at least one of B/A, C/A and D/A ratios is compared with that of an abrasive of known production conditions, determined by XRD analysis, to determine the fluorine content and roasting temperature.

[0029] It is found, as described earlier, a cerium-based abrasive having lower B/A and D/A ratios leaves less scratches on the ground surface. Therefore, the production conditions for an abrasive of required grinding characteristics, e.g., fluorine content and roasting temperature, can be simply determined, by determining in advance the relationship for an abrasive of known production conditions (e.g., fluorine content and roasting temperature) between XRD-determined B/A or D/A ratio and these conditions and comparing these ratios of the abrasives with each other. A cerium-based abrasive can be produced under the fluorine content and roasting temperature thus simply determined. For example, a cerium-based abrasive will leave few scratches on the ground surface, when fluorine content and roasting temperature are controlled for the abrasive in such a way that it satisfies the relationship $B/A<0.06$. The abrasive preferably satisfies $B/A \le 0.05$, more preferably $B/A \le 0.03$, still more preferably $B/A \le 0.01$ to reduce scratches left on the ground surface. For D/A ratio, the production conditions are controlled to give the relationship $D/A<0.04$, preferably $D/A \le 0.03$, more preferably $D/A \le 0.008$ for the same reason.

[0030] For C/A ratio, on the other hand, it is found that there is an adequate range, below which orange peel tends to occur to adversely affect the grinding, and above which grinding force tends to decline, both being undesirable. Therefore, the production conditions for an abrasive with required grinding characteristics, e.g., fluorine content and roasting temperature, can be simply determined, by determining in advance the relationship for an abrasive of known production conditions (e.g., fluorine content and roasting temperature) between the XRD-determined C/A ratio and these conditions and comparing the ratio of the abrasives with each other. A cerium-based abrasive can be produced under the fluorine content and roasting temperature simply determined. A cerium-based abrasive will be an excellent abrasive, and hence desirable, when it satisfies the relationship $0.05 \le C/A \le 0.60$, because it controls formation of orange peel and has a sufficiently high grindability.

[0031] Controlling a fluorine content of the abrasive and roasting temperature based on the two values of B/A or D/A and C/A ratios can give a cerium-based abrasive which leaves few scratches on the ground surface and has a required grindability or higher. More concretely, a fluorine content and roasting temperature are controlled to produce a cerium-based abrasive in such a way that the abrasive has a B/A ratio of less than 0.06 and, at the same time, C/A ratio which gives a required grindability.

[0032] The method of the present invention produces a cerium-based abrasive under the conditions of fluorine content and roasting temperature, controlled to give the abrasive having the required XRD-determined intensity ratios, e.g., B/A ratio. The XRD analysis to determine the intensity ratios such as B/A is used for the quality examination, as described earlier. The quality examination is carried out routinely. It is advantageous when the quality examination results can be used for setting or adjusting the production conditions, because the additional grinding test or the like is no longer necessary. As described above, the XRD-aided quality examination is much more simple than the grinding

to determine weight loss, by which cut thickness was estimated.

[0046] Scratches were evaluated by transmission and reflection. More concretely, the ground surface was irradiated with light from a halogen lamp (300,000 lux) to observe the glass surface, to evaluate the scratches by the extent (size and the numbers) thereof, scored by deducting points from 100 points. The results are given in Table 1.

[0047] Each abrasive was measured for its specific surface area and degree of cohesion. Specific surface area was measured for the accurately weighed sample by an automatic specific surface area analyzer (Manufactured by Yuasa Ionics Co., Ltd., Multisorb 12). Degree of cohesion was measured by a powder tester (Hosokawa Micron Co., Ltd.), for which 355, 250 and 44 μm sieves were used. The results are given in Table 1.

[0048] With the use of X-ray diffractometer MXP18 (Manufactured by MAC Science Co., Ltd.), Abrasives 1 to 10 were analyzed by XRD to determine XRD intensity, with a Cu target and Cu-Kα_1 line as the X-ray source under-the conditions of tube voltage: 40 kV, tube current: 150 mA, diffraction angle (2θ) range: 5 to 80° , sampling width: 0.02°, and scanning rate: 4°/minute. The results are given in Table 2. The XRD analysis data of Abrasives 1 and 6 are given in Figures 2 and 3.

[0049] The XRD analysis measured intensity A of the maximum peak "a" and its diffraction angle θ_A in a diffraction angle (2θ) range from 5 to 80°, diffraction angle θ_B and intensity B of the maximum peak having a diffraction angle in a range of 27.5±0.3° and smaller than the angle of the peak "a," diffraction angle θ_c and intensity C of the maximum peak having a diffraction angle in a range of 26.5±0.5°, and diffraction angle θ_D and intensity D of the maximum peak having a diffraction angle in a range of 24.2±0.5°. The results are given in Table 2, where peak intensities B, C and D are relative to that of peak intensity A (100). As described earlier, the peak is defined as the one whose intensity is 0.5% or more of the peak intensity A, and the peak intensities B, C and D are regarded as zero when they are lower than 0.5% of the peak intensity A.

[0050] The relationship between grindability and C/A ratio is shown in Figure 1.

Table 2

	XRD intensity and intensity ratio						
	C	A	B	D	C/A	B/A	D/A
Abrasive 1	8	100	0	0	0.08	0.00	0.00
Abrasive 2	7	100	0	0	0.07	0.00	0.00
Abrasive 3	50	100	0	0	0.50	0.00	0.00
Abrasive 4	47	100	0	0	0.47	0.00	0.00
Abrasive 5	36	100	6	4	0.36	0.06	0.04
Abrasive 6	34	100	17	10	0.34	0.17	0.10
Abrasive 7	8	100	0	0	0.08	0.00	0.00
Abrasive 8	10	100	0	0	0.10	0.00	0.00
Abrasive 9	53	100	0	0	0.53	0.00	0.00
Abrasive 10	3	100	0	0	0.03	0.00	0.00

[0051] As shown in Table 1, Abrasive 10, having a specific surface area exceeding 12 m^2/g, is a poor abrasive, because of its insufficient properties of very low grindability and high adhesion to the surface to be ground, although good in grinding evaluation.

[0052] Abrasives 1 to 9, having a specific surface area of 12 m^2/g or less, have a correlation between the C/A ratio and grindability, as shown in Tables 1 and 2 and Figure 1. In other words, grindability tends to increase as the C/A ratio increases, and grindability can be estimated for grinding from the C/A ratio.

[0053] It is found, as shown in Tables 1 and 2, that Abrasives 1 to 4 and 7 to 9 out of Abrasives 1 to 9 which have a specific surface area of 12 m^2/g or less but 1.0 m^2/g or more have zero peak intensities B and D, and hence zero B/A and D/A ratios, leaving few scratches on the ground surface. On the other hand, scratches are produced rapidly in a B/A or D/A region of B/A≥0.06 or D/A≥0.04 (Abrasives 5 and 6). This conceivably results from increased fluorine content to further grow the LnOF phase (e.g., LaOF phase), which, although increasing grindability, leaves the LnF_3 phase (e.g., LaF_3 phase) because of the limited growth of the LnOF phase (e.g., LaOF phase) at the normal roasting temperature (around 600 to 1100°C). Abrasives 5 and 6 have higher B/A and D/A ratios but slightly lower C/A ratio than Abrasives 3 and 4.

[0054] It is therefore judged that a cerium-based abrasive leaves less scratches on the ground surface as its B/A or D/A ratio decreases, and, conversely, leaves more scratches when its B/A or D/A is in a region of B/A≥0.06 or D/A≥0.04. Grindability of an abrasive can be judged based on a C/A ratio.

[0055] A cerium-based abrasive leaving only a limited number of scratches on the ground surface can be produced by adjusting its fluorine content and temperature at which it is roasted, based on the B/A or D/A ratio. It will leave few scratches, when its fluorine content and temperature at which it is roasted are adjusted in such a way to avoid B/A≥0.06 or D/A≥0.04.

[0056] Moreover, a cerium-based abrasive having a given grindability or more can be produced when its fluorine content and temperature at which it is roasted are adjusted based on the C/A ratio.

[0057] Therefore, a cerium-based abrasive leaving few scratches on the ground surface and having a given grindability or more can be produced, when its fluorine content and temperature at which it is roasted are adjusted based on B/A or D/A and C/A ratios. More concretely, it is recommended to adjust fluorine content and roasting temperature in such a way to avoid B/A≥0.06 or D/A≥0.04, and, at the same time, to keep a C/A ratio corresponding to a required grindability.

[0058] A cerium-based abrasive can guarantee that it leaves few scratches on the ground surface, when it satisfies B/A<0.06 or D/A<0.04. The abrasive preferably satisfies B/A≤0.05, more preferably B/A≤0.03, still more preferably B/A≤0.01 to reduce scratches left on the ground surface, or D/A≤0.03, more preferably D/A≤0.008 for the same reason.

[0059] A cerium-based abrasive can be sufficiently serviceable, when it simultaneously satisfies the relationships B/A<0.06 or D/A<0.04 and 0.05≤C/A≤0.60, because it leaves few scratches on the ground surface and secures a grindability of around 23 to 40 µm or more as shown in Tables 1, 2 and Figure 1. A cerium-based abrasive simultaneously satisfying the relationships B/A<0.06 or D/A<0.04 and 0.10≤C/A≤0.60 is suitable for primary grinding of glass for liquid crystal and hard disks, because it leaves few scratches on the ground surface and secures a grindability of around 23 to 40µm or more. A cerium-based abrasive simultaneously satisfying the relationships B/A≤0.01 or D/A≤0.008 and

8. The cerium-based abrasive according to Claim 4, **characterized in that** $B/A \leq 0.01$ and $0.05 \leq C/A \leq 0.10$.

9. The cerium-based abrasive according to Claim 5, **characterized in that** $D/A \leq 0.008$ and $0.10 \leq C/A \leq 0.60$.

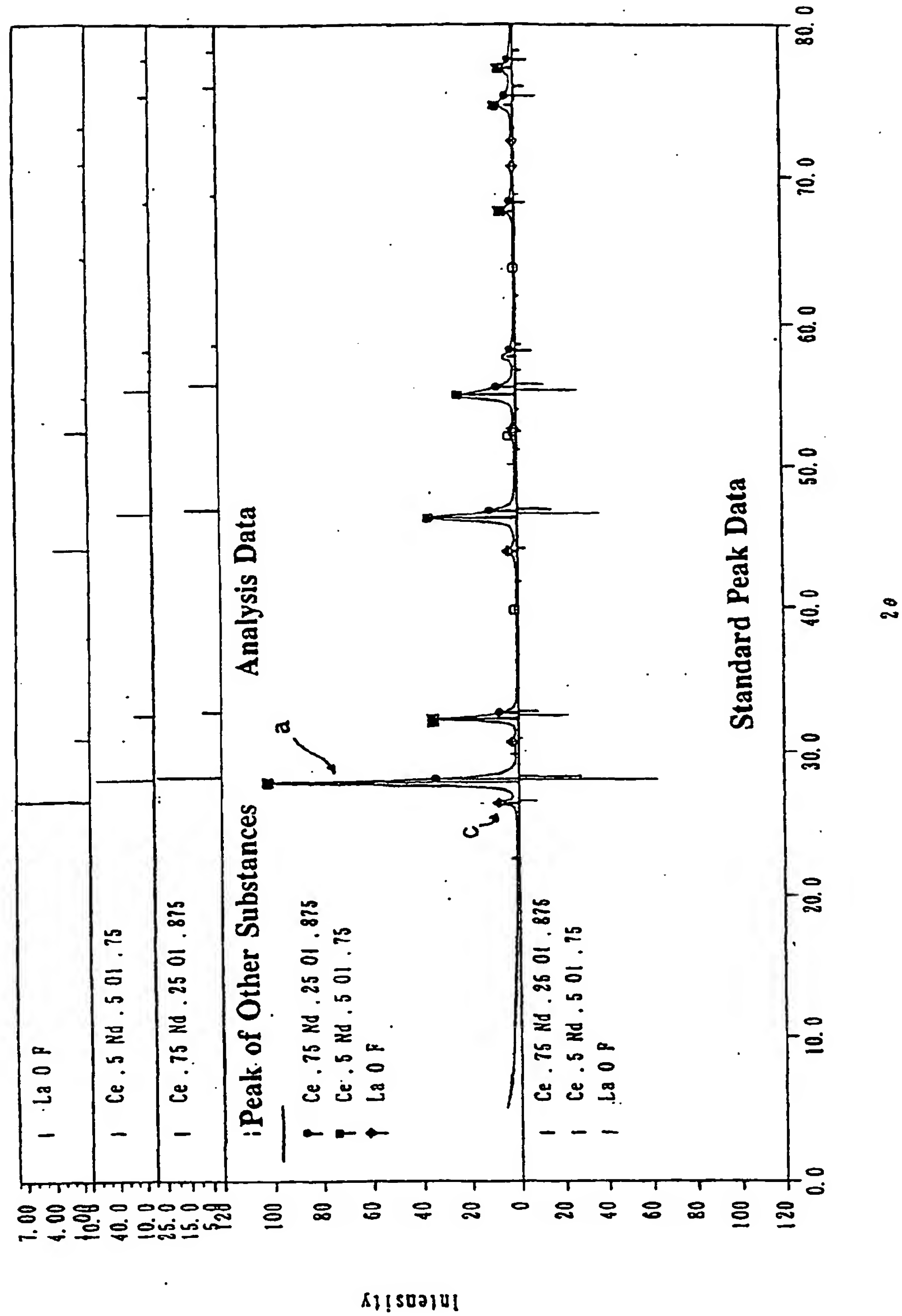
10. The cerium-based abrasive according to Claim 5, **characterized in that** $D/A \leq 0.008$ and $0.05 \leq C/A \leq 0.10$.

11. A method of producing a cerium-based abrasive containing fluorine, and also La or Nd each at 0.5% or more per 100% of Ce, all by atom, and having a specific surface area of 12 m^2/g or less, involving a fluorination step prior to a roasting step,

wherein a fluorine content in the fluorination step and temperature for the roasting step are determined by comparing at least one of intensity ratios B/A, C/A and D/A, determined by XRD analysis with Cu-$K\alpha_1$ line as an X-ray source of a cerium-based abrasive, with that of an abrasive of known grinding characteristics determined by XRD analysis, intensity A being that of the maximum peak "a" in a diffraction angle (2θ) range from 5 to 80°, intensity B being that of the maximum peak having a diffraction angle in a range of 27.5±0.3° and smaller than the angle of the peak "a," intensity C being that of the maximum peak having a diffraction angle in a range of 26.5±0.5°, and intensity D being that of the maximum peak having a diffraction angle in a range of 24.2±0.5°.

12. The method of producing a cerium-based abrasive according to Claim 11, wherein the fluorine content in the fluorination step and the temperature for the roasting step are controlled in such a way to have the abrasive satisfying the relationship $B/A<0.06$ or $D/A<0.04$.

FIG. 2


La O F
Ce .5 Nd .5 O1 .75
Ce .75 Nd .25 O1 .875
Peak of Other Substances
Analysis Data
Standard Peak Data
a
c
Intensity
2θ

INTERNATIONAL SEARCH REPORT

International application No. PCT/JP01/08087

A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl[7] C09K3/14, C01F17/00, B24B37/00, H01L21/304

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int.Cl[7] C09K3/14, C01F17/00, B24B37/00-37/04, H01L21/304

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 11-269455 A (Mitsui Mining & Smelting Co., Ltd.), 05 October, 1999 (05.10.99), Claims (Family: none)	1-12
A	JP 2000-26840 A (Toray Industries, Inc.), 25 January, 2000 (25.01.00), Claims (Family: none)	1-12
A	JP 9-183966 A (Seimi Chemical Co., Ltd.), 15 July, 1997 (15.07.97), Claims (Family: none)	1-12
A	JP 2000-188270 A (Hitachi Chemical Co., Ltd.), 04 July, 2000 (04.07.00), Claims (Family: none)	1-12
A	JP 10-106993 A (Hitachi Chemical Co., Ltd.), 24 April, 1998 (24.04.98), Claims (Family: none)	1-12

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
- "A" document defining the general state of the art which is not considered to be of particular relevance
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Date of the actual completion of the international search	Date of mailing of the international search report
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